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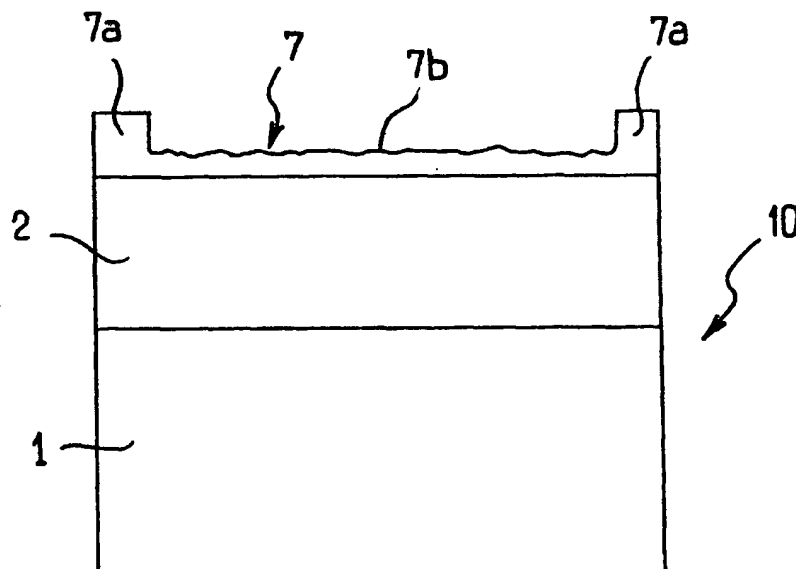
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(54) Title: **MECHANICAL RECYCLING OF A WAFER COMPRISING A BUFFER LAYER, AFTER HAVING TAKEN A LAYER THEREFROM**



(57) Abstract: Method of recycling a donor wafer (10) after taking off at least one useful layer, the donor wafer (10) comprising successively a substrate (1), a buffer structure (I) and, before taking-off, a useful layer. The method comprises employing mechanical means to remove part of the donor wafer (10) on the side where the taking-off took place, such that, after removal of substance, there remains at least part of the buffer structure (I) capable of being reused as at least part of a buffer structure (I) during a subsequent taking-off of a useful layer. The present document also relates to: a) methods of taking off a thin layer from a donor wafer (10) which can be recycled according to the invention; b) donor wafers (10) which can be recycled according to the invention.



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“MECHANICAL RECYCLING OF A WAFER COMPRISING A BUFFER
LAYER, AFTER HAVING TAKEN A LAYER THEREFROM”

The present invention relates to the recycling of a donor wafer comprising a buffer layer after transfer of a thin semiconductor layer from the donor wafer to a receiving substrate.

5 The term “buffer layer” generally refers to a transition layer between a first crystalline structure such as a substrate and a second crystalline structure having the prime function, of modifying properties of the material, such as structural or stoichiometric properties or atomic surface recombination properties.

10 In the particular case of a buffer layer, the latter may make it possible to obtain a second crystalline structure, the lattice parameter of which differs substantially from that of the substrate.

To this end, the buffer layer may have a composition which varies gradually with thickness, the gradual variation of components of the buffer layer then being directly associated with a gradual variation of its lattice parameter.

15 It may also have a more complex form such as a variation in composition with a variable rate, a sign inversion of the rate or discontinuous jumps in composition, possibly completed with a constant composition layer for containing defects.

20 Mention is then made of a metamorphic (buffer) layer or of a metamorphic embodiment, such as a metamorphic epitaxy.

Produced on the buffer layer, a layer or a superposition of layers may be taken off from the donor wafer in order to be transferred to a receiving substrate, in order to produce a particular structure.

25 One of the major applications of transferring thin layers formed on a buffer layer relates to the formation of strained silicon layers.

A layer is made of a material which is “strained” in tension or in compression if its lattice parameter in the interface plane is respectively greater or less than its nominal lattice parameter.

30 Otherwise, a layer is said to be made of a “relaxed” material if the latter is substantially close to its nominal lattice parameter, a nominal lattice parameter being the lattice parameter of the material in its bulk form in equilibrium.

When a layer is made of silicon strained in tension, some properties, such

as the electron mobility of the material, are clearly improved.

Other materials, such as for example SiGe, may also be subject to a substantially similar taking-off.

5 The transfer of such layers onto a receiving substrate in particular by a process called Smart-cut®, and known to a person skilled in the art, then makes it possible to produce structures such as SOI (Semiconductor On Insulator) structures.

For example, after taking off a layer of relaxed SiGe, the structure obtained may then act as a support for growing silicon.

10 Since the nominal lattice parameter of SiGe (dependent on the germanium content) is greater than the nominal lattice parameter of silicon, growth of silicon on the SGOI (Silicon-Germanium On Insulator) pseudo-substrate obtained makes it possible to provide the silicon layer strained in tension.

As an illustration, an example of such a process is described in the IBM document by L.J. Huang *et al.* ("SiGe-On-Insulator prepared by wafer bonding and layer transfer for high-performance field-effect transistors", Applied Physics Letters, 26/02/2001, vol. 78, No. 9) in which a method of producing an Si/SGOI structure is presented.

15 Another example of such a process is given in the document US 2002/007481.

Other applications of metamorphic growth are possible, especially with semiconductors of the III-V family.

Thus, transistors are commonly produced using GaAs-based or InP-based technologies.

25 In terms of electron performance, InP has a substantial advantage over GaAs, in particular, a combination of an InP layer and an InGaAs or InAlAs layer makes it possible to improve electron mobilities.

However, the ability to market components using InP technology is limited faced with GaAs technology, particularly in terms of cost, availability, mechanical weakness and the size of bulk substrates (the maximum diameter for InP typically being 4 inches compared with 6 inches for GaAs).

30 A solution to this problem seems to be found with reference to a receiving substrate, an InP layer removed and obtained by metamorphic epitaxy of a buffer layer on a GaAs substrate.

Certain taking-off processes, such as a process of the "etch-back" type, then lead to destruction of the remaining part of the substrate and of the buffer layer during taking-off.

In some other taking-off processes, such as a Smart-cut® process, the substrate is recycled but the buffer layer is lost.

However, the metamorphic production technique is complex.

Optimizing and producing such a buffer layer may therefore involve a lengthy, difficult and expensive operation.

Furthermore, internal strains due to the variations in composition may cause the appearance of a high rate of crystalline defects, such as dislocations and point defects.

These internal strains, and therefore the generation of defects, may be minimized in particular by increasing the thickness over which the lattice parameter varies.

It is mainly for this reason that the buffer layers usually produced are thick, with a typical thickness ranging from one to a few micrometers.

However, economic and technical restraints limit some essential properties of the buffer layer, such as its thickness or a certain structural complexity.

For all these reasons among others, it would be wise to avoid completely forming a buffer layer after each recycling of the substrate.

The present invention intends to achieve this aim by providing, according to a first aspect, a method of recycling a donor wafer after having taken at least one useful layer of a material chosen from semiconductor materials, the donor wafer comprising successively a substrate, a buffer structure and, before taking-off, a useful layer, the method comprising removal of substance on the side of the donor wafer where the taking-off took place, characterized in that the removal of substance comprises employing mechanical means so that, after removal of substance, at least part of the buffer structure remains, this at least part of the buffer structure capable of being reused as a buffer structure during a subsequent useful layer taking-off.

According to a second aspect, the invention provides a method of taking off a useful layer on a donor wafer in order to be transferred to a receiving substrate, characterized in that it comprises:

- (a) bonding the donor wafer to the receiving substrate;
- (b) detaching a useful layer bonded to the receiving substrate from the donor wafer;
- (c) recycling the donor wafer complying with the said method of recycling.

5 According to a third aspect, the invention provides a method of cyclically taking off a useful layer from a donor wafer, characterized in that it comprises several steps of taking off a useful layer, each of these steps complying with the said method of taking-off.

10 According to a fourth aspect, the invention provides an application of the said method of cyclically taking-off or of the said method of taking-off, for producing a structure comprising the receiving substrate and the useful layer, the useful layer comprising at least one of the following materials: SiGe, strained Si, Ge, an alloy belonging to the III-V family, the composition of which is respectively chosen from the possible (Al,Ga,In)-(N,P,As) combinations.

15 According to a fifth aspect, the invention provides a donor wafer having supplied a useful layer by taking-off and capable of being recycled complying with the said method of recycling, characterized in that it successively comprises a substrate and a remaining part of the buffer structure.

20 Other aspects, aims and advantages of the present invention will become more clearly apparent on reading the following detailed description of operating the preferred methods thereof, given by way of non-limiting example and made with reference to the appended drawings in which:

Figure 1 shows a donor wafer according to the prior art.

Figure 2 shows a donor wafer after taking-off.

25 Figure 3 shows a donor wafer after a first recycling step.

Figure 4 shows the various steps of a method according to the invention successively comprising taking-off of a thin layer from a donor wafer and recycling of the donor wafer after taking-off.

30 The main object of the present invention consists in recycling a wafer comprising a buffer structure (i.e. any structure behaving as a buffer layer), after at least one useful layer has been taken off from the wafer so as to integrate this useful layer into a semiconductor structure, the recycling including at least partial recovery of the buffer structure so that it can be reused in a subsequent taking-off.

The said recycling operation must therefore comprise a suitable treatment

which does not damage at least part of the buffer structure.

Indeed, a buffer structure usually contains crystallographic defaults, such as dislocations, which can propagate and increase in size in an important manner when energy is supplied on it, this energy could provide from thermal treatments, chemical process or mechanical process.

For instance, if a buffer structure of SiGe is heated at a temperature of 350°C, 450°C or 550°C, the structural state change with respect to the temperature chosen (see for instance the document "Structural characterisation and stability of Si_{1-x}Gex/Si(100) heterostructures grown by molecular beam epitaxy" of Re et al., in the Journal of Crystal Growth, vol. 227-228, pp. 749-755, July 2001). With the increase of temperature, the buffer structure will tend to decrease its internal stress by relaxing them in slip planes, stacking defaults or other structural relaxation types. This can bring some future difficulties at the interface with the useful layer to be formed. It is then important to keep these internal stress confined in the buffer structure.

Recycling must then be carried out in a manner, with adapted means for recycling, so as to prevent and limit the extension of these crystalline stress inside the buffer structure which can damage its properties and which can thus damage the properties of the useful layer formed on it.

Advantageously, it has a crystallographic structure which is substantially relaxed and/or without a noteworthy number of structural defects on the surface.

A "buffer layer" is as already defined more generally above in this document.

Advantageously, a buffer layer is comprised in the buffer structure and has at least one of the two following functions:

1. decreasing the density of defects in the upper layer;
2. matching a lattice parameter of two crystallographic structures with different lattice parameters.

With regard to the second function of the buffer layer, the latter is an interlayer between the two structures, and around one of its faces it has a first lattice parameter substantially identical to that of the first structure and around its other face, it has a second lattice parameter substantially identical to that of the second structure.

In the rest of this document, the buffer layers or structures described will

in general comply with this latter buffer layer.

However, the present invention also relates to any buffer layer or any buffer structure as defined in this document in the most general manner.

Furthermore, an example of a method according to the invention will be described below, including recycling a donor wafer of a useful layer by taking-off, the donor wafer initially consisting of a support substrate and a buffer structure.

With reference to Figure 1, a donor wafer 10 (donor of a thin layer by taking-off) included in the known prior art consists of a support substrate 1 and a buffer structure I.

The application for this donor wafer 10 in the present invention is that of taking off a useful layer, from the part 4 of the buffer structure I and/or at least part of an overlayer formed on the surface of the buffer structure I (not shown in Figure 1), in order to integrate it into a structure, such as an SOI structure.

The support substrate 1 of the donor wafer 10 comprises at least one semiconductor layer having a first lattice parameter at its interface with the buffer structure I.

In a particular configuration, the support substrate 1 consists of a single semiconductor having the first lattice parameter.

In a first configuration of the buffer structure I, the latter consists of a buffer layer 2.

The buffer layer 2, located on the support substrate 1, in this case makes it possible to present at its surface a second lattice parameter substantially different from the first lattice parameter of the substrate 1, and thus to have, in the same donor wafer 10, two layers 1 and 4 respectively having different lattice parameters.

Furthermore, the buffer layer 2 may make it possible, in some applications, for the overlying layer to prevent the latter from containing a high defect density and/or being subject to noticeable stresses.

Furthermore, the buffer layer 2 may make it possible, in some applications, for the overlying layer to have a good surface condition.

In general, the buffer layer 2 has a lattice parameter which changes gradually with thickness in order to establish the transition between the two lattice parameters.

Such a layer is generally called a metamorphic layer.

This gradual change of the lattice parameter may be produced

continuously within the thickness of the buffer layer 2.

Alternatively, it may be carried out in "stages", each stage being a thin layer with a substantially constant lattice parameter which is different to that of the underlying stage, so as to discretely change the lattice parameter stage by stage.

5 It may have also have a more complex form, such as a variation in composition with a variable rate, a sign inversion of the rate or discontinuous jumps in composition.

The change of the lattice parameter in the buffer layer 2 is advantageously found by increasing therein, starting from the substrate 1, in a gradual manner, the
10 concentration of at least one atomic element which is not contained in the substrate 1.

Thus, for example, a buffer layer 2 produced on a substrate 1 made of a unitary material could be made of a binary, tertiary, quaternary or higher material.

Thus, for example, a buffer layer 2 produced on a substrate 1 made of a
15 binary material could be made of a tertiary, quaternary or higher material.

The buffer layer 2 is advantageously produced by growth on the support substrate 1, for example by epitaxy, using known techniques such as the CVD and MBE techniques (abbreviations for "Chemical Vapour Deposition" and "Molecular Beam Epitaxy", respectively).

20 In general, the buffer layer 2 may be produced by any other known method, in order to obtain, for example, a buffer layer 2 consisting of an alloy of various atomic elements.

A minor step of finishing the surface of the substrate 1 underlying the buffer layer 2, for example by CMP polishing, may possibly precede the
25 production of the buffer layer 2.

In a second configuration of the buffer structure I, and with reference to Figure 1, the buffer structure I consists of a buffer layer 2 (substantially identical to that of the first configuration) and of an additional layer 4.

The additional layer 4 may be between the substrate 1 and the buffer layer
30 1, or on the buffer layer 1, as shown in Figure 1.

In a first particular case, this additional layer 4 may constitute a second buffer layer, such as a buffer layer making it possible to confine defects, and thus to improve the crystalline quality of a layer produced on the buffer structure I.

This additional layer 4 is made of a semiconductor preferably having a

constant material composition.

The choice of the composition and of the thickness of such a buffer layer 4 to be produced are then particularly important criteria to achieve this property.

Thus, for example, the structural defects in an epitaxially grown layer
5 usually decrease gradually within the thickness of this layer.

In a second particular case, the additional layer 4 is located on the buffer layer 1 and functions as an upper layer to the buffer layer 2.

Thus it may fix the second lattice parameter.

In a third particular case, the additional layer 4 is located on the buffer
10 layer 1 and plays a role in the taking-off that will be carried out in the donor wafer 10, such as a taking-off at its level.

The additional layer may also have several functions, such as functions chosen from these last three particular cases.

In an advantageous configuration, the additional layer 4 is located on the
15 buffer layer 2 and has a second lattice parameter different from the first lattice parameter of the support substrate 1.

In a particular case of this latter configuration, the additional layer 4 is made of a material relaxed by the buffer layer 2, and has the second lattice parameter.

20 The additional layer 4 is advantageously produced by growth on the buffer layer 2, for example by epitaxial growth by CVD or MBE.

In a first embodiment, the growth of the additional layer 4 is carried out *in situ*, directly in continuation with the formation of the underlying buffer layer 2, the latter also in this case being advantageously formed by layer growth.

25 In a second embodiment, the growth of the additional layer 4 is carried out after a minor step of finishing the surface of the underlying buffer layer 2, for example by CMP polishing, heat treatment or other smoothing techniques such that the dislocations and other defaults contained in the buffer layer 2 don't propagate, don't increase in size and don't create any slip planes, stacking defaults or other
30 defaults which can decrease the quality of the final buffer structure I thus formed.

The taking-off of a useful layer from the donor wafer 10 is operated according to one of the following main modes:

- (1) the useful layer to be taken off is part of the additional layer 4.
- (2) the useful layer to be taken off is part of an overlayer (not shown in Figure 1)

which has been formed beforehand on the buffer structure I, for example by epitaxial growth possibly preceded by finishing the surface of the buffer structure I.

The donor wafer 10 then functions as a substrate for the growth of the
5 overlayer.

The latter may comprise one or more thin layers depending on the taking-off mode that it is desired to use.

Furthermore, advantageously, it has a lattice parameter substantially identical to that of the relaxed material of the free face of the buffer structure I,
10 such as a layer of an identical material, or another material which would have all or some of its crystallographic structure strained in tension or in compression, or the combination of these two types of material.

In a particular embodiment of the donor wafer 10, one or more interlayers are furthermore inserted between the buffer structure I and the overlayer. In this
15 case, this or these interlayers are not taken off.

(3) the useful layer to be taken off is part of the additional layer 4 and an overlayer (formed in a substantially identical manner to that described in the second taking-off mode).

Whatever the taking-off mode chosen, and with reference to Figure 2,
20 after taking-off and in the majority of cases, projecting parts 7a and/or rough parts 7b appear on the taking-off surface of the remaining donor wafer 10.

This taking-off surface "in relief" belongs to a post-taking-off layer 7 located above the buffer layer 2.

This post-taking-off layer 7 consists of all or some of the layer 4, possibly
25 one or more interlayers and possibly part of an overlayer depending on the taking-off mode chosen from the three previously discussed taking-off modes.

The parts 7a and 7b in relief appearing on the surface of the post-taking-off layer 7 mainly depend on the taking-off mode and on the technique operated during taking-off.

30 • Thus, for example, a taking-off mode currently used in industry consists in taking off the useful layer not over the entire surface of the donor wafer 10, but only over part of the latter (which is generally a substantially centred part) leaving, on the surface of the donor wafer 10, projecting parts, such as those referenced 7a. These projecting parts are generally integral and

located at the periphery of the surface of the donor wafer 10, all the projecting parts then being known in the business as "taking-off ring".

- Thus, for example, known taking-off techniques such as, for example, those that we will study further and later on in this document, such as the Smart-cut[®] technique already mentioned, sometimes cause surface roughness such as that referenced 7b on the taking-off surface.

Once the taking-off is carried out, recycling according to the invention is operated in order to restore the donor wafer 10.

A first step of recycling according to the present invention consists in removing at least the relief parts 7a and 7b (shown in Figure 2).

This removal of substance according to the invention is operated such that, after the removal, at least part of the buffer structure I remains, which can be used again during subsequent taking-off of a new useful layer.

The remaining part of the buffer structure I, after removal of substance, is thus recycled, unlike the known recycling of the prior art.

In a first particular case of recycling, and concerning the said second mode of taking-off (2), it could be advantageous to choose a thickness of the overlayer so that, after taking-off, the remaining part of the overlayer (which is the post-taking-off layer 7) is removed by standard mechanical means for removing substance, such as polishing means or CMP, without removing substance from the safe buffer structure I, and thus preserve the entire buffer structure I.

Thickness of material removed during the recycling by standard mechanical means like polishing is typically of around 2 micrometers, even if current development succeeds to reach thickness around 1 micrometer.

In a second particular case of recycling, and concerning the said second mode of taking-off (2), it could be advantageous to choose a thickness of the overlayer and of the additional layer 4 so that, after taking-off, the remaining part of the overlayer (which is the post-taking-off layer 7) and at least a part of the additional layer 4 is removed by standard mechanical means for removing substance, such as polishing means or CMP, without removing substance from the safe buffer layer 2, and thus preserve the entire buffer layer 2.

A removal of substance comprises operation using means for mechanically attacking substance, such as polishing or grinding.

A polishing technique commonly used consists in placing a donor plate 10

between a polishing head and a polishing plate which is able to rotate about a drive shaft.

The respective main surfaces of the polishing head and of the polishing plate are substantially parallel.

5 A force applied to the polishing head presses the donor wafer 10 against the upper face of the plate.

The rotating movement of the donor wafer 10 with respect to the plate causes friction on one face of the donor wafer 10, and therefore polishes this face.

10 In a preferred mode, the polishing head, accompanied by the donor wafer 10, moves over the upper surface of the polishing plate along a path determined in order to homogenize the polishing as much as possible. This movement may, for example, be a translational to-and-fro movement along a particular axis or a helical movement.

15 The polishing plate is advantageously coated with a textured material or fabric.

A polishing solution making it possible to lubricate the friction action of the plate on the donor wafer may advantageously be injected.

Post-polishing cleaning of the wafer surface, generally with deionized water which is injected, may follow the polishing.

20 Post-polishing rinsing may be operated between the polishing and the cleaning, generally with a solution comprising a suitable surfactant which is injected. The prime function of a surfactant is to disperse the residual particles as much as possible in the rinsing solution, which may continue to erode the surface of the slice, and thus to decrease their deposition on the surface, and allow their
25 removal.

One of more of these solutions are advantageously injected so as to moisten the fabric covering the plate which thus distributes the solution as well as possible over the entire surface of the donor wafer 10.

30 In a first embodiment of the plates, the said plate functions of polishing, rinsing and cleaning are fulfilled only by a single plate.

However, to improve the productivity of the whole method, devices with several plates will be preferred:

In a second embodiment of the plates, the polishing function is fulfilled by a polishing plate and the rinsing and cleaning functions are fulfilled by a single

plate called a rinsing/cleaning plate. This embodiment, which uncouples the polishing from the rinsing/cleaning, improves the quality of the rinsing by using, for the rinsing, a plate which is completely free of any particulate residues which may remain attached to a plate.

5 In a third embodiment of the plates, the polishing plate, the rinsing plate and the cleaning plate are separate plates. This embodiment uncouples, with respect to the second embodiment, the rinsing from the cleaning thus improving the final cleanness of the surface of the slice by using, for the cleaning, a plate which is completely free of any particulate residues which may remain attached to
10 a rinsing plate.

 In addition to the polishing, abrasive particles, such as silica particles, may be involved in order to improve the abrasion of the substance.

 In addition to the polishing, chemical agents may be involved in order to accompany the mechanical attack

15 operated by the polishing plate with chemical etching.

 In an advantageous operational mode of removing substance from the donor wafer 10, chemical-mechanical planarization, also called CMP, is carried out, the principle of which is to bring together the polishing surface of the polishing plate with a polishing fluid comprising abrasive particles and a chemical
20 etching agent.

 In addition to the mechanical polishing, the polishing fluid then jointly uses the chemical etching by using the etching agent and mechanical abrasion by means of abrasive particles, of the surface to be polished of the donor wafer 10.

 Here again, the removal of substance may be followed by rinsing and/or
25 cleaning of the polished surface of the donor wafer 10.

 It should be noted that the rinsing may in some cases act, not only on faster removal of the residual and abrasive particles of the polishing, but also on the chemical action of the polishing.

 This is because, if the chemical etching agent used during polishing has a
30 basic pH, by adding a generally acidic surfactant agent to the polishing solution, fast stopping of the chemical action of the polishing solution is promoted.

 For certain semiconductors, such as silicon, the chemical action predominates over the mechanical action (the abrasive particles used while polishing the surface of such semiconductors being small).

Such rinsing with an acidic surfactant therefore makes it possible, especially for the materials mentioned in the last paragraph, to significantly stop the action of the polishing and to control its effect on the slice. As such, the post-polishing thickness is thus guaranteed and reproducible.

5 Thus it is possible to control the stopping of polishing, and therefore more accurate control of the thickness removed.

Furthermore, progressive injection of the rinsing solution will be preferred: injection which is too fast leads to a rapid decrease in the pH of the polishing solution and may, in some cases of semiconductors, such as silicon, have
10 the consequence of increasing the size of the abrasive particles by agglomeration and therefore exposing it to abrasive damage caused by these larger particle agglomerates.

An example of an operational application of planarizing a layer is presented here in the case where the layer to be planarized comprises, at least in
15 part, silicon.

The solution suitable for polishing silicon is generally a basic solution having a pH of between 7 and 10, and preferably between 8 and 10, the chemical agent preferably then having a nitrogen-containing base, such as ammonia.

The abrasive particles are preferably silica molecules, with sizes of about
20 a tenth of a micron.

If it is decided to rinse, a surfactant having a pH of preferably between 3 and 5, or even around 4, with a CMC (Critical Micellar Concentration) close to at least 0.1%, will be used.

The time of the rinsing step will advantageously be about 50% of the
25 polishing time.

These mechanical or chemical-mechanical means are particularly advantageous within the scope of the invention for controlling the quantity of substance removed so as to allow at least part of the buffer structure I to be preserved.

30 However, in general, the removal of substance from the donor wafer 10 may comprise operating all mechanical means of attacking substance, such as, for example, grinding or bombardment with atomic species.

This removal of substance may possibly be preceded by a heat treatment making it possible to further smooth the surfaces to be removed and/or remove the

projecting parts 7a or the rough parts 7b.

The heat treatment can, for example, be operated as disclosed in the document US 6,596,610, where such projecting parts 7a and rough parts 7b are removed by a heat treatment. This technique is advantageously performed in the case of a taking-off having taken place in an overlayer on the buffer structure, so that this heat treatment mainly acts on this overlayer and not on the buffer structure I, preserving the latter to increasing of inside defaults.

One of the following substance removal modes is therefore used:

- (a) removing part of the post-taking-off layer 7 comprising at least the relief parts 7a and 7b; or
- (b) removing the entire post-taking-off layer 7; or
- (c) removing the entire post-taking-off layer 7 and part of the buffer layer 2.

If the post-taking-off layer 7 comprises part of an original overlayer, the substance removal mode (a) then preferably comprises completely taking off this overlayer part.

With reference to Figure 3, the part of the original buffer structure which remains after substance removal is referenced I'.

It consists of:

- the entire original buffer structure I when the substance removal mode (a) was used and when the latter did not involve taking off any part of the additional layer 4; or
- the buffer layer 2 and part of the additional layer 4 when the substance removal mode (a) was used and when the latter involved taking off part of the additional layer 4; or
- the buffer layer 2 when the substance removal mode (b) was used; or
- part of the buffer layer 2 when the substance removal mode (c) was used.

A second recycling step comprises, after the first recycling step relating to substance removal, reforming at least some of the layers removed during the first step.

First of all, and in certain cases, it will be preferred to finish the surface of the donor wafer 10 where the substance removal operated during the first recycling step took place, so as to remove any roughness which may have appeared during

the substance removal.

To this end, for example, a heat treatment will be used such that the dislocations and other defaults contained in the buffer structure I don't propagate, don't increase in size and don't create any slip planes or stacking defaults, as
5 already discussed.

The second step then involves restoring the buffer structure I from the remaining buffer structure I', when part of the original buffer structure I was removed during the first recycling step.

Advantageously, the restoration of the buffer structure I is such that, once
10 formed, the latter is substantially identical to the original buffer structure I.

However, in a particular embodiment, it will be possible to slightly alter some production parameters in order to obtain a buffer structure I which is slightly different from the original. For example, the concentrations of certain compounds in a material will be slightly altered.

15 Restoring the buffer structure I involves reforming the removed part of the buffer layer 2 when part of the original buffer layer 2 was cut away during the first recycling step.

Restoring the buffer structure I involves reforming all or part of the additional layer 4 when all or part of the original additional layer 4 was cut away
20 during the first recycling step.

In this case, it will be possible to produce an additional layer 4 with a thickness substantially identical to or substantially different from the original.

Once the buffer structure I is restored, an overlayer may possibly be formed above it, which overlayer will at least partly comprise a new useful layer to
25 be removed, possibly with one or more interlayers between the buffer structure I and the overlayer.

The layers possibly formed during this second recycling step are advantageously produced by layer growth on their respective underlying layers, for example by CVD or MBE epitaxial growth.

30 In a first case, at least one of these layers I and 5 is grown *in situ*, directly in continuation with the formation of the underlying growth support, the latter also being formed in this case advantageously by layer growth.

In a second case, at least one of these layers is grown after a minor step of finishing the surface of the underlying growth support, for example by CMP

polishing, heat treatment or other smoothing techniques such that the dislocations and other defaults contained in the buffer structure I don't propagate, don't increase in size and don't create any slip planes, stacking defaults or other defaults which can decrease the quality of the buffer structure I.

5 Thus, a donor wafer 10 which is substantially identical to the original, that is to say the donor wafer 10 shown in Figure 1, is finally obtained, with the exception of modifications desired and carried out by a person skilled in the art.

 The donor wafer 10 obtained in this way comprises at least part of the original buffer structure I, and therefore at least part of the original buffer layer 2,
10 which makes it possible to avoid its complete, lengthy and expensive reformation, as was the case in the known recycling methods.

 With reference to Figures 4a to 4f, the various steps are shown of a method of taking off a thin layer from and of recycling a donor wafer 10 after taking-off according to the invention, which uses a donor wafer 10 with a layer
15 structure substantially identical to that described above with reference to Figure 1 and which therefore comprises, with reference to Figure 4a, a substrate 1, and a buffer structure I.

 In this exemplary method according to the invention, an overlayer 5 has been added above the buffer structure I.

20 The removal that will be carried out during this method will relate to taking-off of the overlayer 5 and possibly of part of the buffer structure I.

 In the same way and in other structural configurations of the donor wafer 10, there may be several overlayers and the taking-off would then relate to the overlayers and possibly part of the buffer structure I, or there may be no overlayer
25 and the taking-off would then relate to only part of the buffer structure I.

 These two layers I and 5 have advantageously been formed by epitaxial growth according to known techniques, for example by CVD and MBE.

 In a first case, at least one of these layers is grown *in situ*, directly in continuation with the formation of the underlying growth support, the latter also
30 being in this case advantageously formed by layer growth.

 In a second case, at least one of these layers is grown after a minor step of finishing the surface of the underlying growth support, for example by CMP polishing, heat treatment or other smoothing techniques, such that the dislocations and other defaults contained in the buffer structure I don't propagate, don't

increase in size and don't create any slip planes, stacking defaults or other defaults which can decrease the quality of the buffer structure I.

A method of taking off a thin layer is shown in Figures 4b and 4c.

5 A first preferred taking-off step of the invention consists in creating a fragile zone in the donor layer 10, in order to carry out a subsequent detachment at this fragile zone, and thus separate the desired useful layer.

Several techniques that can be operated to create such a fragile zone are presented here:

10 A first technique, called Smart-cut®, known to a person skilled in the art (and descriptions of which may be found in a number of works covering techniques for reducing wafers) consists, in its first step, in implanting atomic species (such as hydrogen ions) with a particular energy in order to create in this way a fragile zone.

15 A second technique consists in forming a fragile interface by creating at least one porous layer, as described for example in document EP-A-0 849 788.

The fragile zone advantageously formed according to one of these two techniques is created above the substrate 1:

- in the buffer layer of the buffer structure I; or
 - between the buffer layer and any relaxed layer of the buffer structure I;
- 20 or
- in any relaxed layer of the buffer structure I; or
 - between the buffer structure I and the overlayer 5; or
 - in the overlayer 5 if the latter is thick enough; this is the particular case of an overlayer 5 consisting of a stack of layers.

25 With reference to Figure 4b, a second step relating to taking off a thin layer consists in attaching a receiving substrate 6 to the surface of the overlayer 5.

The receiving substrate 6 forms a mechanical support which is rigid enough to support the overlayer 5 which will be removed from the donor wafer 10, and to protect it from any mechanical strains coming from the outside.

30 This receiving substrate 6 may, for example, be made of silicon or of quartz or of another type of material.

The receiving substrate 6 is attached by placing it in intimate contact with the overlayer 5 and by bonding it thereon, in which molecular adhesion is advantageously carried out between the substrate 6 and the overlayer 5.

This bonding technique, together with variants, is in particular described in the document entitled "Semiconductor Wafer Bonding" (Science and technology, Interscience Technology) by Q.Y. Tong, U. Gösele and Wiley.

If necessary, the bonding is accompanied by a suitable pretreatment of the
5 respective surfaces to be bonded and/or by a supply of heat energy and/or a supply of an additional binder.

Thus, for example, heat treatment applied during or just after the bonding makes it possible to stiffen the bonded connections.

The bonding may also be controlled by a bonding layer, such as silica,
10 inserted between the overlayer 5 and receiving substrate 6, having particularly high molecular bonding abilities.

Advantageously, the material forming the bonding face of the receiving substrate 6 and/or the material of the bonding layer possibly formed, is electrically insulating, in order to produce an SOI structure from the taken off layers, the
15 semiconductor layer of the SOI structure then being the useful layer 5 transferred.

Once the receiving substrate 6 is bonded, part of the donor wafer 10 is taken off at the fragile zone formed beforehand, by detaching it.

In the case of the said first technique (Smart-cut®), in a second step, the implanted zone (forming the fragile zone) is subjected to a heat and/or mechanical
20 treatment, or other supply of energy, in order to detach it at the fragile zone.

In the case of the said second technique, the fragile layer is subjected to mechanical treatment or other supply of energy, in order to detach it at the fragile layer.

Detachment at the fragile zone according to one of these two techniques,
25 for example, makes it possible to remove most of the wafer 10, in order to obtain a structure comprising possibly the rest of the buffer structure 1, the overlayer 5, any bonding layer and the receiving substrate 6.

A step of finishing the surface of the structure formed, at the removed layer, is then advantageously operated in order to remove any surface roughness,
30 inhomogeneities in thickness and/or undesirable layers, by using, for example, chemical-mechanical polishing CMP, etching or a heat treatment.

A post-taking-off layer 7' forms the part of the donor wafer 10 located above the substrate 1 which remains after taking-off, this entire wafer forming a donor wafer 10' to be sent for recycling in order to be reused subsequently during

another layer taking-off.

The recycling steps are shown in Figures 4d, 4e and 4f.

With reference to Figure 4d, a first recycling step corresponds to removing part of the post-taking-off layer 7'.

5 Mechanical or chemical-mechanical abrasion or etching according to one of those already discussed above, is operated to remove part of the post-taking-off layer 7'.

Several techniques for removing substance by various mechanical means may also be operated, especially if the post-taking-off layer 7' comprises several
10 different original layers (for example, part of the overlayer 5 and part of the buffer structure I), such as, for example, making abrasion by CMP and by simple polishing follow one another.

This mechanical attack of substance may be preceded and/or followed by surface treatments, such as chemical etching, heat treatment or smoothing such that
15 the dislocations and other defaults contained in the buffer structure I don't propagate, don't increase in size and don't create any slip planes, stacking defaults or other defaults which can decrease the quality of the buffer structure I.

In all cases, and at the end of this first recycling step, with reference to Figure 4d, at least part of the buffer structure I' remains.

20 With reference to Figures 4e and 4f, a second recycling step corresponds to the restoration of layers which are substantially identical to those which existed before taking-off, with the respective formations of any missing part of the buffer structure I and of an overlayer 5.

The layers are advantageously restored by forming a layer according to a
25 technique which is substantially identical to one of these detailed above.

In a first case, at least one of these layers is grown *in situ*, directly in continuation with the formation of the underlying growth support, the latter in this case also being advantageously formed by layer growth.

In a second case, at least one of these four layers is grown after a minor
30 step of finishing the surface of the underlying growth support, for example by CMP polishing, heat treatment or other smoothing techniques such that the dislocations and other defaults contained in the buffer structure I don't propagate, don't increase in size and don't create any slip planes, stacking defaults or other defaults which can decrease the quality of the buffer structure I.

The layers 1 and 5 obtained of the donor wafer 10''' are not necessarily identical to the layers 1 and 5 of the donor wafer 10, it being possible for the donor wafer shown in Figure 4d to act as a substrate for other types of layers.

After recycling the donor wafer 10 according to the invention, a method
5 of taking off a useful layer can then be operated again.

Thus, in an advantageous context of the invention, a cyclic method of taking off a useful layer from a donor wafer 10 according to the invention is operated, by making the following succeed each other repeatedly:

- a taking-off mode; and
- 10 • a recycling method according to the invention.

Before operating the cyclic taking-off method, it is possible to operate a method of producing the donor wafer 10 according to the invention with one or more of the techniques for producing thin layers on a substrate, described above.

In the remainder of this document, we present examples of configurations
15 of donor wafers 10 comprising buffer structures 1, and capable of being operated by a method according to the invention.

In particular, we will present materials which can advantageously be used in such donor wafers.

As we have seen, a buffer structure 1 produced on a substrate 1 having a
20 first lattice parameter has, most of the time, the prime function of having a second lattice parameter on its free face.

Such a buffer structure 1 then comprises a buffer layer 2 making it possible to produce such matching of a lattice parameter.

The technique most often employed to obtain a buffer layer 2 having this
25 property is to have a buffer layer 2 consisting of several atomic elements comprising:

- at least one atomic element which is in the composition of the substrate 1; and
- at least one atomic element, none or very little of which is in the
30 substrate 1, having a concentration changing gradually within the thickness of the buffer layer 2.

The gradual concentration of this element in the buffer layer 2 will be the main cause of the gradual change of the lattice parameter in the buffer layer 2, in a metamorphic manner.

Thus, in this configuration, a buffer layer 2 will mainly be an alloy.

The atomic elements chosen for the composition of the substrate 1 and for the buffer layer 2 may be of type IV, such as Si or Ge.

For example, in this case, it is possible to have a substrate 1 made of Si
5 and a buffer layer 2 made of SiGe with a Ge concentration changing progressively with thickness between a value close to 0 at the interface with the substrate 1 and a particular value on the other face of the buffer layer 2.

In another scenario, the compositions of the substrate 1 and of the buffer layer 2 consist of an alloy of the III-V family, such as the possible
10 (Al,Ga,In)-(N,P,As) combinations.

The buffer layer 2 preferably consists of an alloy which is of ternary type or of a higher degree.

For example, in this case, it is possible to have a substrate 1 made of AsGa and a buffer layer 2 comprising As and/or Ga with at least one other
15 element, the latter element changing progressively with thickness between a value close to 0 at the interface with the substrate 1 and a particular value on the other face of the buffer layer 2.

The composition of the substrate 1 and of the buffer layer 2 may comprise pairs of atomic elements of type II-VI, such as the possible (Zn,Cd)-(S,Se,Te)
20 combinations.

Below, we offer some examples of such configurations:

Example 1: After recycling, the donor wafer 10 consists of:

- a substrate 1 made of Si;
- 25 - a buffer structure I made of SiGe with a buffer layer 2 and an additional layer 4;
- a post-taking-off layer 7 made of Si or of SiGe which forms the rest of an overlayer 5 after taking off part of the latter.

30 These donor wafers 10 are particularly used when taking off layers of SiGe and/or of strained Si in order to produce SGOI, SOI or Si/SGOI structures.

The buffer layer 2 preferably has a Ge concentration progressively increasing from the interface with the substrate 1, in order to make the SiGe lattice parameter change as explained above.

The thickness is typically between 1 and 3 micrometers in order to obtain good structural relaxation at the surface, and to contain the defects associated with the difference in lattice parameter so that they are buried.

5 The additional layer 4 is made of SiGe relaxed by the buffer layer 2, with a Ge concentration which is advantageously uniform and substantially equal to that of the buffer layer 2 near their interface.

The concentration of germanium in the silicon within the additional SiGe layer 4 is typically between 15% and 30%.

10 This limitation at 30% represents a typical limitation of the current techniques, but may be made to change in the next few years.

The additional layer 4 has a thickness which may vary hugely depending on the case, with a typical thickness of between 0.5 and 1 micron.

Example 2: After recycling, the donor wafer 10 consists of:

- 15
- an Si substrate 1;
 - a buffer structure I with an SiGe buffer layer 2 and an additional layer 4 of substantially relaxed Ge;
 - a post-taking-off AsGa layer 7 which forms the rest of an overlayer 5 after taking off part of the latter.

20

The buffer layer 2 preferably has a Ge concentration increasing progressively from the interface with the substrate 1, in order to make the lattice parameter change between that of the Si substrate 1 and that of the additional Ge layer 4.

25 To this end, in the buffer layer 2, the Ge concentration is made to progress from about 0 to about 100%, or more precisely around 98%, for complete agreement of the theoretical lattice of the two materials.

Example 3: After recycling, the donor wafer 10 consists of:

- 30
- a substrate 1 comprising at least one AsGa part at its interface with the buffer structure I;
 - a buffer structure I made of a III-V material;
 - a post-taking-off layer 7 comprising a III-V material which constitutes the rest of an overlayer 5 after taking-off of part of the latter.

The prime benefit of this buffer structure I is to match the lattice parameter of the material V of the overlayer 5 (whose nominal value is about 5.87 angströms) to that of the AsGa (whose nominal value is about 5.65 angströms).

In the bulk III-V materials, and by comparing bulk InP to bulk AsGa, the latter is less expensive, more widely available on the semiconductor market, less fragile mechanically, a material from which the use of technologies with contact by a rear face is better known, and whose size may reach high values (typically 6 inches instead of 4 inches for bulk InP).

In a particular configuration of the donor wafer 10 before taking-off, the overlayer 5 before taking-off comprised InP to be removed.

Since the bulk InP has a dimension generally limited to 4 inches, the donor wafer 10 gives, for example, a solution to producing an InP layer dimensioned at 6 inches.

A buffer structure I for producing such an overlayer requires a thickness typically greater than one micron, and which will be made to change towards greater thicknesses, especially if it can be recycled according to the present invention.

The epitaxial growth technique usually operated to produce such a buffer structure I is furthermore particularly difficult and expensive, it is therefore beneficial to be able to recover it at least partially after taking off the useful layer.

Advantageously, the buffer structure I comprises a buffer layer 2 consisting of InGaAs with an In concentration changing between 0 and about 53%.

The buffer structure I may further comprise an additional layer 4 made of a III-V material, such as InGaAs or InAlAs, with a substantially constant concentration of the atomic elements.

In a particular taking-off case, the InP overlayer 5 and part of the additional layer 4 will be removed in order to transfer it to a receiving substrate.

Thus it will be possible to profit from any electrical or electronic properties existing between the two removed materials.

This is the case, for example, if the part of the additional layer 4 removed is made of InGaAs or of InAlAs: electronic band discontinuities between the latter material and InP create improved electronic mobilities in the taken off layers.

Other configurations of the donor wafers 10 are possible, comprising other III-V compounds, such as InAlAs or the like.

Typical applications of such layer taking-off are HEMT or HBT ("High-Electron Mobility Transistor" and "Heterojunction Bipolar Transistor",
5 respectively) production.

In the semiconductor layers presented in this document, other components may be added to them, such as carbon with a carbon concentration substantially less than or equal to 50% or more particularly with a concentration less than or equal to 5% in the layer in question.

10 Finally, the present invention is not limited to a buffer structure I, an interlayer 8 or an overlayer 5 made of materials presented in the examples above, but extends also to other types of alloys of IV-IV, III-V, II-VI type.

It should be specified that these alloys may be binary, ternary, quaternary or of a higher degree.

15 The present invention is not limited either to a recyclable buffer layer 2 or buffer structure I having the prime function of matching the lattice parameter between two adjacent structures with different respective lattice parameters, but also relates to any buffer layer 2 or buffer structure I as defined in the most general manner in the present document and which can be recycled according to the
20 invention.

The structures finally obtained after taking-off are not limited either to SGOI or SOI structures.

CLAIMS

1. Method of recycling a donor wafer (10) after having taken at least one useful layer of a material chosen from semiconductor materials, the donor wafer (10) comprising successively a substrate (1), a buffer structure (I) and, before taking-off, a useful layer, the method comprising removal of substance on the side of the donor wafer (10) where the taking-off took place, characterized in that the removal of substance comprises employing mechanical means so that, after removal of substance, at least part of the buffer structure (I) remains, this at least part of the buffer structure (I') capable of being reused as a buffer structure (I) during a subsequent useful layer taking-off .
2. Method of recycling a donor wafer (10) according to the preceding claim, characterized in that the operation of mechanical means when removing substance comprises polishing.
3. Method of recycling a donor wafer (10) according to one of the preceding claims, characterized in that the operation of mechanical means when removing substance comprises abrasive polishing.
4. Method of recycling a donor wafer (10) according to the preceding claim, characterized in that the operation of mechanical means when removing substance is accompanied by chemical etching.
5. Method of recycling a donor wafer (10) according to one of the preceding claims, characterized in that the operation of mechanical means when removing substance comprises chemical-mechanical planarization.
6. Method of recycling a donor wafer (10) according to one of the preceding claims, characterized in that the operation of mechanical means is preceded and/or is followed by a surface smoothing treatment.
7. Method of recycling a donor plate (10) according to the preceding claim, characterized in that the surface smoothing treatment comprises a heat treatment

such that the dislocations and other defaults contained in the buffer structure (1) don't propagate, don't increase in size and don't create some slip planes, stacking defaults or other defaults which can decrease the quality of the buffer structure (1).

5 8. Method of recycling according to one of the preceding claims, characterized in that, before taking-off, the buffer structure (1) comprises a buffer layer (2) and an additional layer (4), the additional layer (4) having:

- a thickness which is great enough to confine defects; and/or
 - a surface lattice parameter which is substantially different from that
- 10 of the substrate (1).

9. Method of recycling according to the preceding claim, characterized in that the donor wafer (10) comprises:

- a substrate (1) consisting of Si;
- 15 — a buffer structure (I) comprising an $\text{Si}_{1-x}\text{Ge}_x$ buffer layer (2) with a Ge concentration x increasing with thickness between 0 and a y value, and an $\text{Si}_{1-y}\text{Ge}_y$ layer (4) relaxed by the buffer layer (2).

20 10. Method of recycling according to one of the preceding claims, characterized in that the removal of substance comprises the removal of part of the buffer structure (I) remaining after the taking-off.

25 11. Method of recycling according to Claims 8 and 10, characterized in that the removal of substance comprises the removal of at least part of the additional layer (4) remaining after taking-off.

12. Method of recycling according to claims 8 and 10, characterized in that the removal of substance comprises the removal of part of the buffer layer (2).

30 13. Method of recycling according to one of the preceding claims, characterized in that, before taking-off, the donor wafer (10) comprised an overlayer (5) which comprised the useful layer to be removed, and in that the removal of substance comprises, after taking-off, the removal of the remaining overlayer (5).

14. Method of recycling according to the preceding claim, characterized in that the thickness of the overlayer (5) has been chosen so that, after taking-off, standard mechanical means for removing substance, such as polishing means, can be operated on this overlayer (5) during the removal of substance, without removing substance from the buffer structure (I).

15. Method of recycling according to the claims 8 and 6, characterized in that the thickness of the overlayer (5) and the thickness of the additional layer (4) have been chosen so that, after taking-off, standard mechanical means for removing substance, such as polishing means, can be operated on this overlayer (5) and on this additional layer (4) during the removal of substance, without removing substance from the buffer layer (2).

16. Method of recycling according to one of the three preceding claim combined with claim 9, characterized in that the overlayer (5) comprises SiGe and/or strained Si.

17. Method of recycling according to one of the claims 13 to 15 combined with claim 9, characterized in that $y=1$, and in that the overlayer (5) comprises AsGa and/or Ge.

18. Method of recycling according to one of the preceding claims, characterized in that it further comprises, after the step of removing substance from the donor wafer (10), a step of forming layer on the side of the donor wafer (10) where the removal of substance took place, so as regenerating the donor wafer (10).

19. Method of recycling according to the preceding claim and one of the claims 10 to 12, characterized in that the step of forming layer comprises an operation of forming a new part of the buffer structure (I) above the remaining part of the buffer structure (I').

20. Method of recycling according to one of the two preceding claims and to

one of the claims 13 to 15, characterized in that the step of forming layer comprises an operation of forming an overlayer (5) on the donor wafer (10) so as to form at least one new useful layer to be subsequently taken off.

5 21. Method of recycling according to one of the three preceding, characterized in that the layer(s) is formed during the step of forming layer by crystal growth.

22. Method of recycling according to one of the preceding claims,
10 characterized in that the donor wafer (10) comprises at least one layer further comprising carbon with a carbon concentration in the layer which is substantially less than or equal to 50%.

23. Method of recycling according to one of the preceding claims,
15 characterized in that the donor wafer (10) comprises at least one layer further comprising carbon with a carbon concentration in the layer which is substantially less than or equal to 5%.

24. Method of taking off a useful layer on a donor wafer (10) in order to be
20 transferred to a receiving substrate (6), characterized in that it comprises:

- (a) bonding the donor wafer (10) to the receiving substrate (6);
- (b) detaching a useful layer bonded to the receiving substrate (6) from the donor wafer (10);
- (c) recycling the donor wafer (10) complying with the method of
25 recycling according to one of the preceding claims.

25. Method of taking off a useful layer according to the preceding claim, characterized in that it comprises, before step (a), a step of forming a bonding layer.
30

26. Method of taking off a useful layer according to one of the two preceding claims, characterized in that:

- it further comprises, before step (a), a step of implanting atomic species through the surface of the donor wafer (10) neighbouring the buffer

- 29 -

structure (I) at a determinate depth, in order to form a fragile zone at this depth; and in that:

- the step (b) is operated by supplying energy to the donor wafer (10) in order to detach a structure comprising the receiving substrate (6) and the useful layer at the fragile zone.

27. Method of taking off a useful layer according to one of the claim 24, characterized in that :

- it further comprises, before step (a), a step of forming by porosification a layer in the donor wafer (10) followed by a growth of a layer (which will become the useful layer after detaching of step (b)), the porosified layer forming a fragile zone inside or above the buffer structure (I); and in that :
- the step (b) is operated by supplying energy to the donor wafer (10) in order to detach a structure comprising the receiving substrate (6) and the useful layer at the fragile zone level.

28. Method of taking off a useful layer according to one of Claims 24 to 30, characterized in that the useful layer detached during step (b) comprises part of the buffer structure (I).

29. Method of taking off a useful layer according to one of Claims 24 to 27, characterized in that the donor wafer (10) comprises, before taking-off, an overlayer (5) located on the side away from the substrate (1), and in that the useful layer detached during step (b) comprises at least part of the overlayer (5).

30. Method of cyclically taking off a useful layer from a donor wafer (10), characterized in that it comprises several steps of taking off a useful layer, each of these steps complying with the method of taking-off according to one of Claims 24 to 29.

31. Application of the method of cyclically taking-off according to one of the preceding claim or of the method of taking-off according to one of Claims 24 to

29, for producing a structure comprising the receiving substrate (6) and the useful layer, the useful layer comprising at least one of the following materials:

SiGe, strained Si, Ge, an alloy belonging to the III-V family, the composition of which is respectively chosen from the possible (Al,Ga,In)-(N,P,As) combinations.

5

32. Application of the method of cyclically taking-off method according to the Claim 30 or of the method of taking-off according to one of Claims 24 to 29, for producing semiconductor-on-insulator structure, a such structure comprising the receiving substrate (6) and the useful layer.

10

33. Donor wafer (10) having supplied a useful layer by taking-off and capable of being recycled complying with the method of recycling according to one of Claims 1 to 23, characterized in that it successively comprises a substrate (1) and a remaining part of the buffer structure (I).

1/3

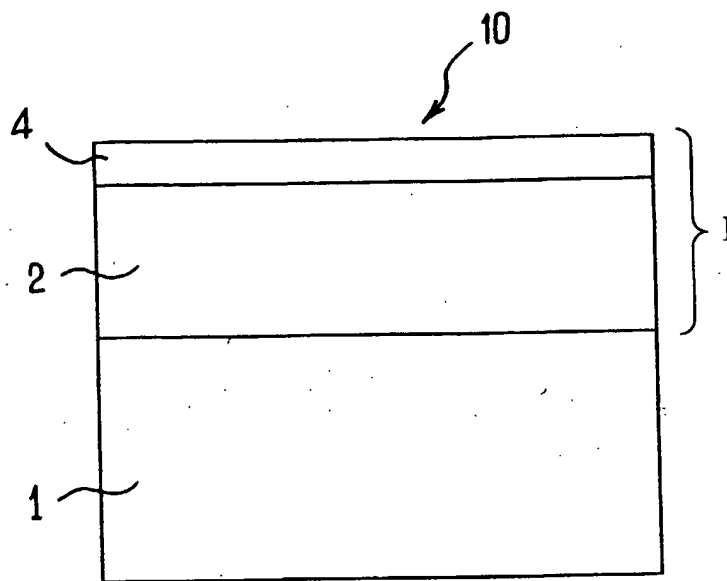


FIG.1

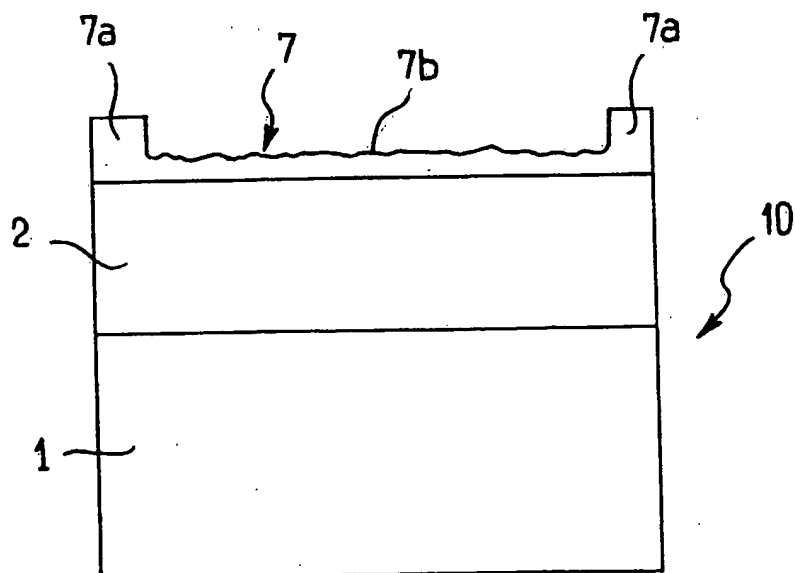


FIG.2

2 / 3

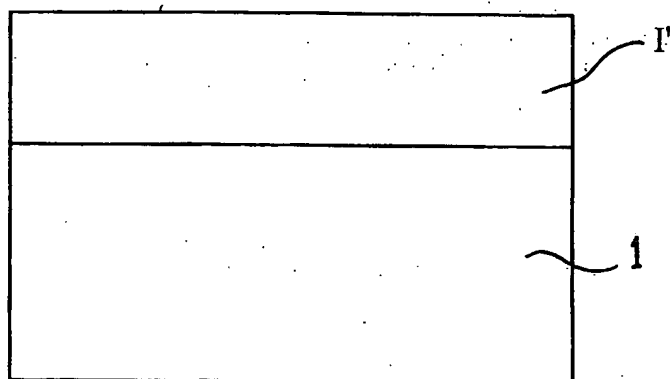


FIG.3

3 / 3

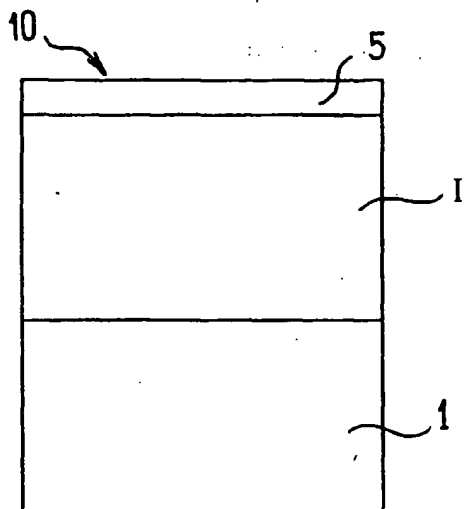


FIG. 4a

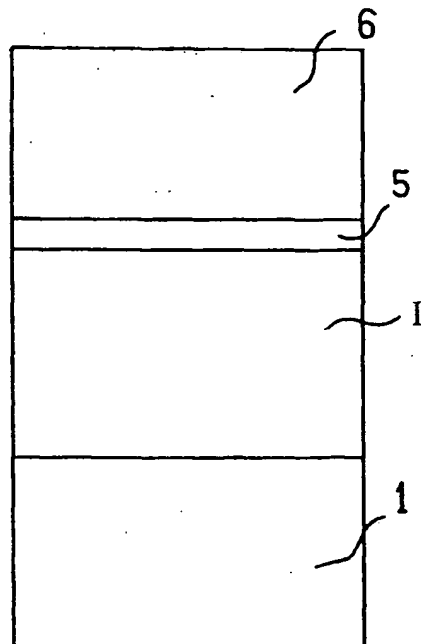


FIG. 4b

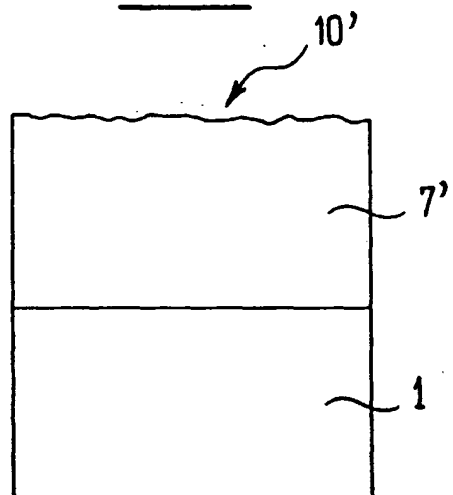


FIG. 4c

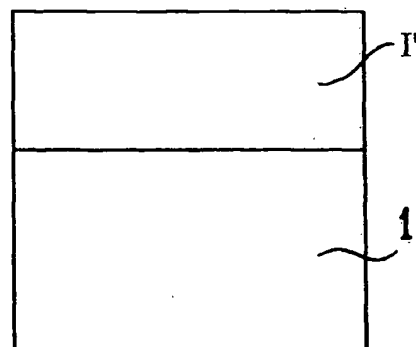


FIG. 4d

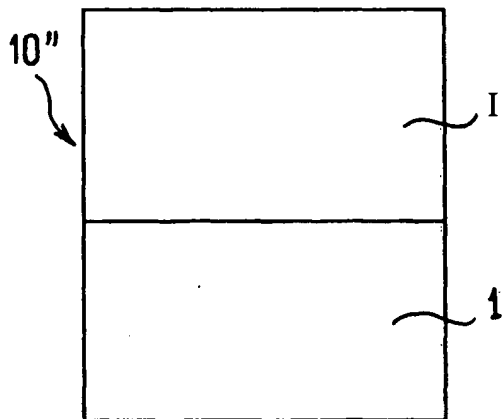


FIG. 4e

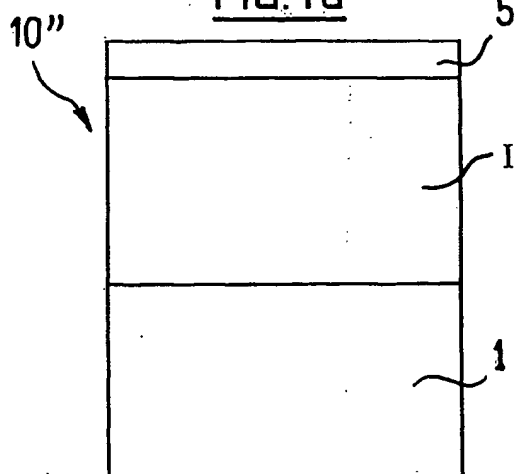


FIG. 4f